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12 INVENTION PATENT APPLICATION A1

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56 - List of documents mentioned in the research report: *See end of this brochure.*

80 - Reference to other national documents connected:

71. Applicant (s): ASSOCIATION POUR LA VALORISATION DES DEJECTIONS ANIMALES (ASSOCIATION FOR APPRECIATION OF ANIMAL EXCREMENT) Association set up under Law of 1901 - France

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54 - Procedure for treatment and appreciation of animal excrement obtained in off-soil industrial stockbreeding operations, such as liquid manures.

57 The invention refers to a procedure for treatment and appreciation of animal excrements, such as liquid manures, and is characterised by consisting of the following stages:

- Reception (1) of these excrements in a storage tank;
- Flocculation (2) of essentially the fermentescible material contained in these excrements;
- Separation (3) of the essentially fermentescible materials from the essentially non-fermentescible materials in order to obtain an essentially organic solid S_1 and a residual water E_1;
- Precipitation (4) of the nitrates and phosphates contained in said residual water E_1, in the form of magnesium ammonia phosphate in order to obtain an essentially mineral solid S_2 and a fairly non-contaminated residual water E_2;
- Separation (5) of this essentially mineral solid S_2, precipitated from the fairly non-contaminated residual water E_2.

RECEPTION

CATIONIC FLOCCULATION

SEPARATION OF FERMENTESCIBLE MATTER

PRECIPITATION OF AMMONIA IONS AND PHOSPHATES

ANIONIC FLOCCULATION

SEPARATION

Procedure for treatment and appreciation of animal excrements obtained in off-soil industrial stockbreeding installations, such as manures.

The agricultural economy of industrialised countries has for many years focussed on the intensive stockbreeding of animals for meat production or reproduction. The concept of intensive stockbreeding as an attempt to meet the great market requirements has revolutionised production techniques and led to practising stockbreeding outside traditional natural areas in order to control the parameters involved in the animals' growth or reproduction more easily. This type of stockbreeding operation, known as off-soil, is now very common in both fowl and swine or bovine sectors.

Off-soil industrial stockbreeding, though entailing much higher production figures than the ones obtained through traditional stockbreeding techniques, does not however cease to pose a number of problems, one of which is the production of excrements. Indeed, these stockbreeding techniques have contributed to the modification of the compositions of animal excrements, due to the evolution of the nutrients used, but above all to the localised concentration of these excrements.

The problems involved by the concentratio.n of animal origin excrements are increasing particularly in industrial swine stockbreeding operations. In spite of being an excellent fertiliser, liquid manure is nevertheless a source of environmental problems to the extent that it cannot be fully absorbed by this form of appreciation.

The main disadvantage meant by liquid manures lies in the smell that these give off, which may hinder or prevent the implementation of units in the proximity of urban centres. Liquid manure's micro-organism content constitutes a possible source of bacteriological contamination.

The liquid nature of this manure and its physical-chemical composition have led to using spreading on farmland around the production units as a channel for appreciation. This technique enables a very considerable improvement in the fertility of the soils treated. Nevertheless, the amounts of materials thus distributed can exceed the soils' acceptance capacities and lead in the longer or shorter term to disastrous consequences for the environment.

Pig manure is a close mixture of faeces, bristles and reflux of food. The manure can also contain rainwater supplied in its composition during storage. The volume of manure produced by a pig depends essentially on the animal's age and its type of diet. On average, each pig produces 1 m^3 of manure, which leads in intensive industrial operations to average amounts of 2.5 m^3 per place

more easily usable manures, several solutions have been put forward, such as modifying the nature of the food given to pigs reared. In particular, a proposal has been made to reduce the phosphate content of such food in order to obtain a manure less rich in this compound. Nevertheless, this type of method quickly reveals its limits due to the requirements in the field of foodstuffs for animals.

Table 1 shows the average contents in dry materials, total hydrogen and phosphorus of the manures of gestating sows and piglets.

Parameters	Gestating sows	Weaned piglets
Dry material g/l	10.4	8.9
Total nitrogen g/l	6.5	6.3
P_2O_5 g/l	6.5	5.6

Table 1

The amounts of phosphorus and nitrogen are distributed unevenly between the solid and liquid phases of the manure. The average samples of swine manure thus show that 87% of total phosphorus is present in the solid phase while 85 % of total nitrogen is present in the liquid phase.

The physical-chemical composition of the manure varies during the storage phase: organic materials are degraded and the percentage of mineral material increases in respect of the dry material; the amount of ammonia nitrogen increases in respect of the amount of total nitrogen.

Spreading manures specifically involves problems of surface and underground water contamination. Studies have shown that the losses through lixiviation of nitrogen under diverse forms could vary from 5% for spreading in spring to 25% for spreading in autumn or winter. In areas where manure spreading is a highly common practice, spreading operations have also been shown to contribute to the worsening of the phenomenon of atmospheric contamination. These studies have given rise to a regulation of spreading operations, in particular according to seasons, in order to prevent these from causing an excessive increase in the rates of nitrates in the environment and bothersome smells.

The appreciation of manure as fertiliser thus has certain limits due to its own nature, but also due to the amounts produced, which are often extremely large. Although swine stockbreeders often also work as farmers, they do not generally have sufficient farmland to take all the manure

manage these surpluses, manure banks have been created to facilitate regional and inter-regional exchanges between manure producers – stockbreeders – and users - farmers. Nevertheless, the amounts of manure "exported" continue to be random and depend on the receivers' agreement to accept them.

Since these excrements are a source of environmental nuisances, in particular as regards smell and microbiology, diverse means have been sought to eliminate or transform the surplus amounts of manure or to purify these.

The first means that can be used to get rid of these surpluses consists in destroying them by heat. Nevertheless, the destruction of these materials implies a consumption of power and possible atmospheric contamination.

The competent authorities in the sectors of agriculture and the environment have over recent years fostered the development of other techniques in order to exhaust and/or economically appreciate the surplus of manures.

These appreciation techniques have to meet strict regulations as regards dumping in the environment and allow obtaining a residue as rich as possible in fertiliser elements (nitrogen, potassium, phosphorus) in order to give this residue any great commercial value. Their implementation has required a thorough study of the manure composition.

One well-known technique for manure transformation consists in fermenting this in order to extract a gas which can be used for producing steam, electricity or hot water. By this method, the manure goes through a first methane fermentation stage in an anaerobic medium. This fermentation stage leads to obtaining gas. The residue stemming from this fermentation then undergoes for example, centrifuging in order to obtain a liquid phase and a solid phase. The liquid phase can be purified by biological means, while the solid phase undergoes heat drying which leads to obtaining a dry pulverulent residue containing the initial fertilising elements of the manure. This technique has the advantage of obtaining odourless solid and liquid residues. Nevertheless, the power produced in the form of gas does not cover the requirements needed for the evaporation and drying stages, meaning that the treatment is power-deficient. This technique, based mainly on biological procedures, also requires bulky and costly installations.

Another technique considered for treating surpluses of manure consists in causing the evaporation of the water contained in the manure using a carrier fluid consisting of paraffin. This technique consists in acidifying manure at a pH close to 5 in order to make the ammonia that this

can be condensed and the liquid obtained biologically purified. The solid phase obtained consists of dehydrated raw manure.

This technique for heat treatment of liquid manure involves the disadvantage of consuming a lot of power and requiring large-scale installations. It also requires the use of paraffin whose full recycling is not guaranteed, which contributes to increasing its cost. What is more, the dry residue obtained at the end of the process is not deodorised, so it can imply problems for usage and constitute a source of bother.

The purpose of this invention is to put forward a non-destructive procedure for treatment and appreciation of animal excrements obtained from industrial off-soil stockbreeding operations, such as liquid manure, which does not involve the disadvantages implied by present technology's versions. In particular, one of the aims of the invention is to propose a non-destructive procedure for treatment and appreciation of animal excrements for obtaining a residual water without most of the phosphorus, nitrogen and suspended material such as micro-organisms, in particular.

The invention also has the aim of proposing a procedure which can be implemented both in the structure of localised - mobile for example - units grouping a large number of producers.

One of the particular objectives of the invention is to provide a procedure consuming little power, at the same time as ensuring the production of easily appreciable sub-products.

Another objective of the invention is to describe a procedure like this one not resorting to any treatment micro-contaminant and which does not entail the production of any new chemical product.

The invention consists in a procedure for treatment and appreciation of animal excrements obtained in the industrial off-soil stockbreeding operations, such as manure. According to the invention, the procedure is characterised by consisting of the following stages:

- Reception of these excrements in a storage tank;
- Floceculating essentially the fermentescible material contained in said excrements;
- Separation of the floceculated essentially fermentescible materials in order to obtain an essentially organic solid S_1 and a wastewater E_1;
- Precipitating the ammonia ions and phosphates contained in this wastewater E_1 in the form of magnesium ammonia phosphate, in order to obtain an essentially mineral solid S_2 and a fairly non-contaminated wastewater E_2;
- Separation of said essentially mineral solid S_2 precipitated from the fairly non-

$$Mg^{2+} + NH_4^{+} + PO_4^{3-} \rightarrow MgNH_4PO_4$$

According to STUMM and MORGAN, who have studied the conditions for precipitation of magnesium ammonia phosphate in water with no ionic force, $MgNH_4PO_4$ precipitation is favoured in an alkaline solution and the product of solubility of this compound reaches its minimum level at pH = 10.7. According to the mass action law, this reaction needs to be conducted in the presence of a surplus ammonia or phosphorus.

The essentially organic solid S_1 obtained during the procedure can be used by the farmer as an amendment on farmland.

The solid S_2, almost exclusively mineral, is not fermentescible. It contains the essential part of the phosphorus and nitrates of the manure and can be used as a fertiliser in zones where this is lacking or act as a base component in synthesis fertilisers. Its low humidity content makes it pelletable, silo-storable and granulable.

This stage, consisting in flocculating the essentially fermentescible material contained in the excrements, is preferably performed by adding a flocculate in the form of cationic polymer.

Said cationic polymer is also preferably chosen from the group made up of polyethylenamines and polyvinylamines.

Advantageously, said stage consisting in separating the essentially mineral solid from the fairly non-contaminated wastewater E_2, is immediately preceded by a second flocculation stage.

Similarly advantageously, said second flocculation stage is performed by adding a flocculant in the form of an anionic polymer to the residual water E_1.

Although the use of any reagent able to act as a source of magnesium and any source of phosphorus in order to meet the law of mass action to produce the reaction to get the precipitation of the magnesium ammonia phosphate may be considered, it is of particular interest to have said precipitation stage done by adding, in a basic medium, a magnesium sulphate or oxide and phosphoric acid with cadmium content removed.

This magnesium ammonia phosphate precipitation stage is preferentially performed in the presence of lime.

The wastewater E_2 can be used for irrigating farmland or, in another also interesting way, the procedure may include a complementary stage consisting in purifying said fairly non-

These separation stages are preferably done on a draining table or in a press filter. It should be stressed that within the framework of installations for centralised implementation of the invention, these separation stages could be performed by other media, for example, by centrifuging.

The invention also consists of an installation for implementing the procedure characterised in that it includes a single draining table, used for proceeding to the separation stages of both the essentially organic solid S_1 of the wastewater E_1 and that of the essentially mineral solid S_2 of the fairly non-contaminated wastewater E_2.

The invention will be understood better thanks to the following description of non-limitative examples of embodiments of the invention, connected with the drawings, in which:

- Figure 1 represents the flow diagram of the procedure for treatment and appreciation of animal excrements obtained from industrial stockbreeding operations according to the invention, in the structure of a localised treatment unit;
- Figure 2 represents a diagram of the procedure principle according to the invention, which includes a procedure for purification of the wastewaters obtained in order to allow these to be returned to nature, in the structure of a centralised treatment unit.

Example 1

In relation to Figure 1, the procedure according to the invention can be done at the level of a localised unit for treatment and appreciation of the manure placed at the disposal of a limited number of stockbreeders. This unit can for example be mobile and sent from farm to farm.

The process involved in this procedure, for example for a ton of liquid manure, is as follows. The manure is stored (1) in a tank. The composition of the raw manure is shown in Table 2:

Parameters	Amounts
Phosphates (P_2O_5 g/kg)	2.95
Nitrogen ($N.NH_4^+$ g kg)	6.11
Total nitrogen (N g kg)	9.25
COD g kg	84
Dry material g kg	91

An amount of a cationic flocculant agent - polymer Z 78 FS40 marketed by the company BETZ Industrie S.A.- is added to the manure. The polymer is diluted at 2 per thousand in mains

done by adding a diluted solution of the polymer to the manure at a proportion of 20 ml for each 100 g of the initial mixture.

After the flocculation is complete, the mixture goes through a separation stage (3) to obtain an essentially organic solid phase S_1, which contains the fermentescible materials and a residual water E_1. The separation is done on a draining table with a porosity of about 400 µm.

Processing a ton of manure produces 380 kg of solid S_1. This solid phase S_1 obtained from the separation can be taken advantage of by the producer as fertiliser material or can be stabilised for composting and or incorporated as commercial fertiliser formulation.

The wastewater E_1 has a nitrogen content of 4.53 g l and a P_2O_5 phosphorus content of 1.49 g l. This wastewater is then taken to a reactor to subject it to a stage for precipitation (4) of the magnesium ammonia phosphate in a basic medium.

The precipitation reaction takes place in the presence of an excess of magnesium and an excess of phosphorus. The source of magnesium used is magnesia (magnesium oxide) and the source of the phosphorus is phosphoric acid H_3PO_4 with low cadmium content. 32 kg of phosphoric acid corresponding to 7.5 kg of phosphorus, 10 kg of quicklime CaO allowing the pH of the medium to be set at 9.2, and 19 kg of magnesium oxide are added to the residual water E_1. All these reagents can be easily purchased on the market. The pH of 9.2 used means that the amount of lime to be added to the medium can be reduced, at the same time as allowing the precipitation reaction of the magnesia ammonia phosphate to take place. One can opt for using another compound other than the quicklime to increase the pH of the medium, such as overburned lime, potassium or soda. One can also envisage the use of other sources of magnesium, such as magnesium chloride, and other sources of phosphorus, such as potassium hydrogen-phosphate or dipotassium phosphate, industrial phosphorous residues or sludges from a biological dephosphating station, particularly in the cases of tertiary chemical dephosphating.

The precipitation reaction (4) is followed by a flocculation stage (6) intended to foster the separation of the magnesium ammonia phosphate precipitated from the medium. This flocculation stage (6) is implemented through a flocculant presented in the form of anionic polymer.

Greater advantages are obtained by adding the set of reagents slowly, especially in order to avoid the appearance of foam.

The mixture obtained after flocculation can easily be separated into an essentially mineral solid mixture S_2 and a fairly non-contaminated residual water E_2. This last separation stage (5)

Parameters	Amounts
Phosphates (P_2O_5 mg l)	50
($P.PO_4$ mg l)	22
Nitrogen ($N.NH_4^+$ g l)	0.4 to 0.8
Nitrogen total (N g l)	1.02
COD g kg	15 to 28

Table 3

The essentially mineral solid S_2 is composed mainly of magnesium ammonia phosphate. Its ammonia nitrogen content, total nitrogen and phosphate, is stated in Table 4.

Parameters	Amounts
Nitrogen ($N.NH_4^+$ g/kg)	10.1
Total Nitrogen (N g/kg)	12.3
Phosphorus PO_4 g/kg	22.9

Table 4

Hence, the content in fairly non-contaminated residual water E_2 has an ammonia nitrogen and phosphates content almost 90% lower than the initial manure content. This wastewater can easily be reused on farms and, as part of the procedure itself, be used for making dilutions of the flocculant polymers. The solid S_2 constitutes an appreciated product which can for example be used as fertiliser. This solid can be collected and used for example, outside the surplus zone or be transformed in order to be presented in the form of granulate products and be kept a long time, since it contains little organic material, which increases its scope of application.

Example 2

Although it can be reused in the framework of a farming operation, the wastewater E_2 is still too contaminated to be poured into a natural medium. In the case of fixed centralised units where a large number of stockbreeders regularly bring their manure production, this waste water cannot have any direct use and must consequently be purified. In connection with Figure 2, the procedure

a flocculation and a separation process allowing a fairly non-contaminated residual water E_2 and an essentially mineral solid S_2 to be obtained.

The fairly non-contaminated residual water E_2 then undergoes a biological treatment to perfect the purification by extracting the residual oxidisable (COD) contents from this, as well as any nitrogen and phosphorus that this might still contain. This stage of activated sludges (7) is done by making the wastewater E_2 flow off towards an aeration tank, where it goes through alternating nitrifying and denitrifying stages. This process leads to a solid S_3 and a wastewater E_3, with a COD 40 times lower than that of the waste water E_2 and which can then go through a lagooning (pond purification) stage (8) consisting in leaving this waste water to stand in an appropriate zone for a sufficiently long period of time to allow natural elimination of most of the nitrogen and phosphorus still remaining in the effluent. The effluent S_4 obtained from this lagooning (pond purification) stage has an extremely low nitrogen content, of around 0.02 kg m^{-3}, a phosphorus content of around 0.002 kg m^{-3} and a COD of about 0.12 kg m^{-3}. It can be poured into the natural medium.

The examples described herein cannot under any circumstances be considered as limiting the invention. The introduction of a great many modifications could be considered without departing from the framework of the invention. In particular, other flocculants can be used, as well as other magnesium or phosphoric reagents or other compounds to alkalinise the medium.

We should point out that the invention involves the advantage of reducing the germs contained in the treatment outflow, particular the wastewater E_2, since this has a pH of around 9 or above, not favourable for the development of flora.

It has also been observed that the procedure reduces smells, above all through removing the fermentiscible matter at source, thanks to the ammonia sieving in the magnesium ammonia phosphate precipitation stage and thanks to the pH being kept around 9.2, which reduces the separation and evaporation of the ammonia (the pK of NH_3 and NH_4^+ is around 9.2).

CLAIMS

1.- Procedure for treatment and appreciation of animal excrements obtained from industrial off-soil stockbreeding, such as liquid manure, characterised in that this consists of the following stages:

- Reception (1) of these excrements in a storage tank;
- Flocculation (2) of essentially the fermentescible material contained in these excrements;
- Separation (3) of the essentially fermentescible materials from the essentially non-fermentescible materials in order to obtain an essentially organic solid S_1 and a residual water E_1;
- Precipitating (4) the ammonia ions and phosphates contained in said residual water E_1, in the form of magnesium ammonia phosphate so as to obtain an essentially mineral solid S_2 and a fairly non-contaminated wastewater E_2;
- Separation (5) of this essentially mineral solid S_2, precipitated from the fairly non-contaminated wastewater E_2.

2.- Procedure for treatment and appreciation of animal excrements according to claim 1, characterised in that said stage (2), consisting in flocculating essentially the fermentescible material contained in these excrements, is performed by adding a flocculant presented in the form of cationic polymer to said excrements.

3.- Procedure for treatment and appreciation of animal excrements according to claim 2, characterised in the fact that said cationic polymer is selected from a group made up of polyethylenamines and polyvinylamines.

4.- Procedure for treatment and appreciation of animal excrements according to one of claims 1 to 3, characterised by the fact that said stage (5), consisting in separating the essentially mineral solid from the fairly non-contaminated residual water E_2, is immediately preceded by a second flocculation stage.

5.- Procedure for treatment and appreciation of animal excrements according to claim 4, characterised in that said second flocculation stage (6) is done by adding a flocculant presented in the form of an anionic polymer to said residual water E_2.

6.- Procedure for treatment and appreciation of animal excrements according to one of

7.- Procedure for treatment and appreciation of animal excrements according to one of claims 1 to 6, characterised by the fact that said magnesium ammonia phosphate precipitation stage (4) is done in the presence of lime.

8.- Procedure according to either of claims 6 or 7, characterised by the fact that the basic medium is kept at a pH of roughly 9.2.

9.- Procedure according to claim 6, characterised by the fact that phosphoric acid with cadmium removed is fully or partially replaced by potassium hydrogen-phosphate or dipotassium phosphate, industrial phosphorous residues or sludges from a biological dephosphating station.

10.- Procedure for treatment and appreciation of animal excrements according to one of claims 1 to 9, characterised by the fact that this includes a complementary stage consisting in purifying said fairly non-contaminated wastewater E_2 by means of a treatment known as activated sludges (7) and/or by means of lagooning (pond purification) (8).

11.- Procedure for treatment and appreciation of animal excrements according to one of claims 1 to 10, characterised by the fact that said separation stages (3,5) are performed on a draining table and/or in press and/or centrifuging filters.

12.- Installation for the application of a procedure according to claim 11, characterised by including a single separation installation used for proceeding to said separation stages (3,5), on one hand, of the essentially organic solid S_1 of the wastewater E_1 and on the other hand, of the essentially mineral solid S_2 of the fairly non-contaminated wastewater E_2.

13.- Use of the solid and/or liquid wastes stemming from the procedure according to any one of claims 1 to 11 as amendment products for farmland.

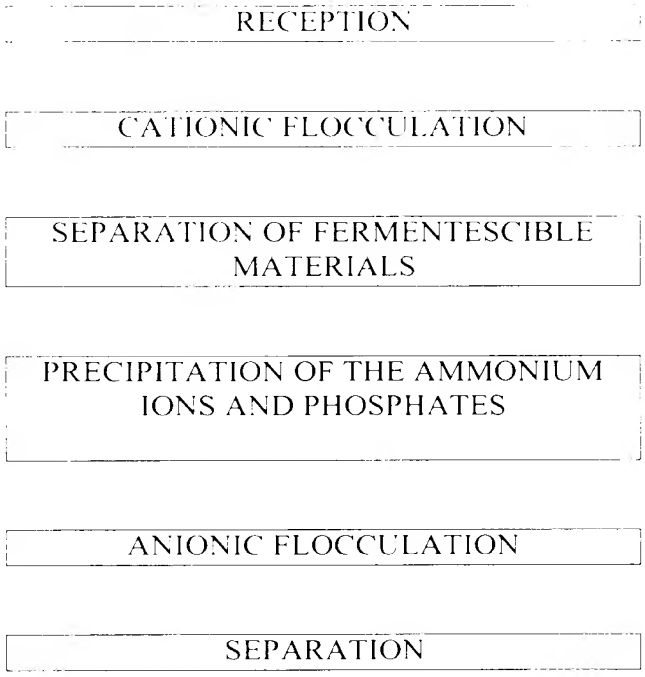
RECEPTION
CATIONIC FLOCCULATION
SEPARATION OF FERMENTESCIBLE MATERIALS
PRECIPITATION OF THE AMMONIUM IONS AND PHOSPHATES
ANIONIC FLOCCULATION
SEPARATION

Fig. 1

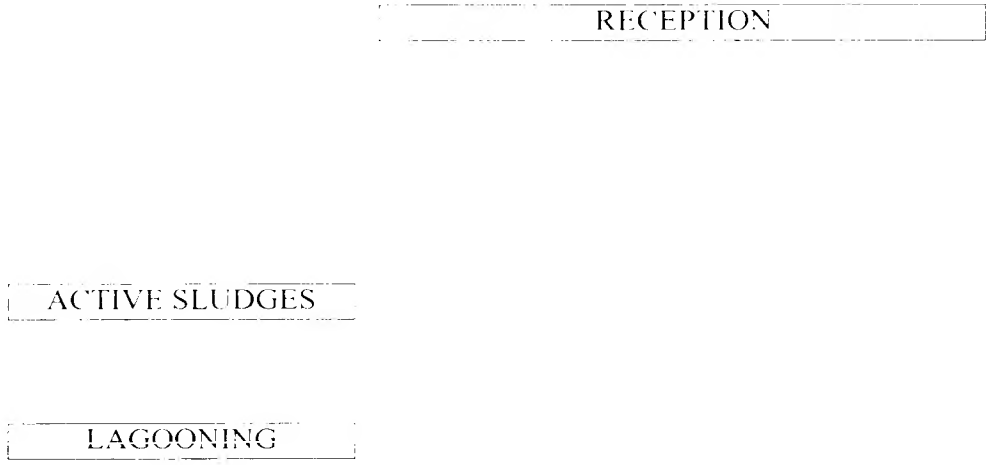

Fig.2

FRENCH REPUBLIC
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National Register N°

RESEARCH REPORT

Established based on the latest claims
registered before starting the research

FR 9201258
FA 467452

DOCUMENTS CONSIDERED RELEVANT		Claims enquired about from the application examined	
Category	Mention of the document with statement, where necessary of the relevant parties		
Y	FR-A-2 414 485 (BAEK ENGINEERING) * claims 1, 4, 9, 12, 14, 15 * * page 2, line 8 – page 4, line 17 *	1,2,4, 10, 11, 13	
Y	PATENT ABSTRACTS OF JAPAN Vol. 012 n° 405 (C-539), 26th October 1988 & JP-A-63 144 UUU (EBARA INFILCO CO LTD) * abbreviated *	1,2,4, 10, 11, 13	
A	DE-A-3 732 896 (SCHULZE-RETTMER, RAINER) * Claims *	6 9	TECHNICAL FIELDS RESEARCHED (Int. Cl-5) CO5F
	Date research completed 16TH SEPTEMBER 1992	Examiner RODRÍGUEZ FONTAO M.	

CATEGORY OF DOCUMENTS MENTIONED

X: particularly relevant in its own right
Y: particularly relevant in combination with another document of the same category
A: relevant in respect of at least one claim or second general technological background.
O: non-written disclosure
P: document intercalated.

T: theory or basic principle of the invention
E: patent document benefiting from a date prior to the registration date and which has only been published on this date of registration or on a later date.
D: mentioned in the application.
L: mentioned for other reasons

&: member of the same family, corresponding document